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PISTON MANOMETER AS AN ABSOLUTE STANDARD FOR VACUUM-GAGE CALIBRATION IN THE RANGE 2 TO 500 MILLITORR

by Isidore Warshawsky

Lewis Research Center
Cleveland, Ohio 44135

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16. Abstract

A thin disk is suspended, with very small annular clearance, in a cylindrical opening in the baseplate of a calibration chamber. A continuous flow of calibration gas passes through the chamber and annular opening to a downstream high-vacuum pump. The ratio of pressures on the two faces of the disk is very large, so that the upstream pressure is substantially equal to net force on the disk divided by disk area. This force is measured with a dynamometer that is calibrated in place with dead weights. A probable error of ±(0.2 millitorr plus 0.2 percent) is attainable when downstream pressure is known to 10 percent.

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SUMMARY

A thin disk is suspended, with very small annular clearance, in a cylindrical opening in the baseplate of a calibration chamber. A continuous flow of calibration gas passes through the chamber and annular opening to a downstream high-vacuum pump. The ratio of pressures on the two faces of the disk is very large, so that upstream pressure is substantially equal to net force on the disk divided by disk area. This force is measured with a dynamometer that is calibrated in place with dead weights. A probable error of ±(0.2 millitorr plus 0.2 percent) is attainable when downstream pressure is known to 10 percent.

INTRODUCTION

The piston manometer, as a means of calibrating vacuum gages, has been described in reference 1. The history of this technique, with bibliographic references, is summarized in reference 2, which gives details of a particular design for the range 10 to 700 microtorr. Reference 2, which emphasizes the calibration of ion gages, also treats the problems that arise from gas contamination resulting from permeation, sorption, and surface reactions. The present report describes a piston manometer for the range 2 to 500 millitorr, where gas contamination problems are negligible. As a consequence, the accuracy of vacuum-gage calibration is more nearly equal to the accuracy of net-force measurement than it was in the case of the lower-range instrument of reference 2. The present instrument is comparable in range and accuracy with the piston manometer described by Ernsberger and Pitman (ref. 3) in 1955. The design to be described differs from that of reference 3 in that the piston diameter is tripled, the piston is a single thin sheet rather than a cylinder, and the force measurement is by a wire-strain-gage-dynamometer rather than a visually observed helical spring; the dynamometer is cali-

brated in place with a dead weight. A principal use for this design has been the calibration of thermal-conductivity-gages and capacitance-diaphragm gages. The measurement principle also permits continuous recording of a changing bell jar pressure, with slightly reduced accuracy.

An advantage of these manometers is that they are usable at all Knudsen numbers, so that no problem is posed by transition from a free-molecule regime to a continuum regime.

THEORY

Basic Arrangement

Figure 1 illustrates the gage-calibration system and piston manometer. The baseplate of a vacuum chamber carries a calibration ring that is interposed between baseplate and bell-jar cover. Gages to be calibrated are mounted on this ring with all their ports in the same horizontal plane. The baseplate opening, through which upper-chamber evacuation would normally take place, is almost blocked by a thin disk suspended from a force-measuring device (dynamometer). The disk floats freely in the baseplate opening, with very small annular clearance. The ratio of disk (piston) area to annulus area is on the order of 100:1.

Calibration gas enters at the top of the bell jar at a rate controlled by a finely ad-

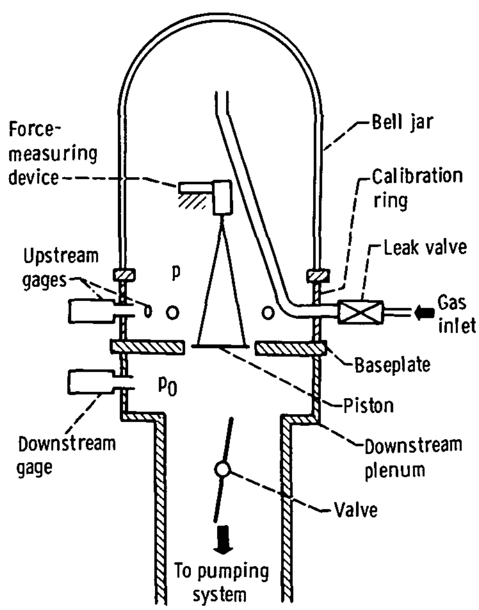

Figure 1. - Basic arrangement of piston manometer for vacuum-gage calibration.

justable leak valve, capable of tight shutoff. Pressure upstream of the leak valve is slightly over 1 bar (1 bar = 10^5 pascals). The gas is directed toward the top of the bell jar in order to facilitate uniform dispersal in the plane of the gage ports. All the entering gas is removed through the annular gap between the knife-edged piston and the cylindrical wall of the opening in the baseplate.

The chamber below the piston is of sufficient volume and cross section to be reasonably isobaric, so that a downstream pressure gage mounted on the chamber wall will measure the pressure on the underside of the piston. A high-vacuum valve separates this lower chamber from the pumping system, which consists of a water-cooled baffle, diffusion pump, and mechanical forepump.

Basic Equations

Steady-state relations. - If piston area is A, net downward force on the piston is ΔF, and upstream and downstream pressures are p and p_0, respectively,

$$p = p_0 + \frac{\Delta F}{A} \tag{1}$$

(Symbols are defined in the appendix.)

If upstream and downstream plenums are at the same temperature and contain the same gas (the calibration gas), conservation of mass requires that

$$(p - p_0)G_a = p_0\dot{V}_0 \tag{2}$$

where $\dot{V}_0$ is the volumetric pumping speed in the downstream plenum and G_a is the annulus conductance, defined as the throughput for unit pressure difference. Throughput is R_0T/M times the mass flowrate, where R_0 is the universal gas constant, T is absolute gas temperature, and M is molecular weight of the gas.

Equation (1) holds even when temperatures are not constant and when mass is not conserved. Equation (2) holds at all Knudsen numbers, although the representation of G_a in terms of annulus dimensions and gas properties will differ for different flow regimes.

In the pressure range of interest in this report, the volumetric pumping speed of most diffusion-pumped systems is not constant, but drops off at higher pressures (as shown in fig. 2, which represents a typical pumping system). Writing equation (2) as

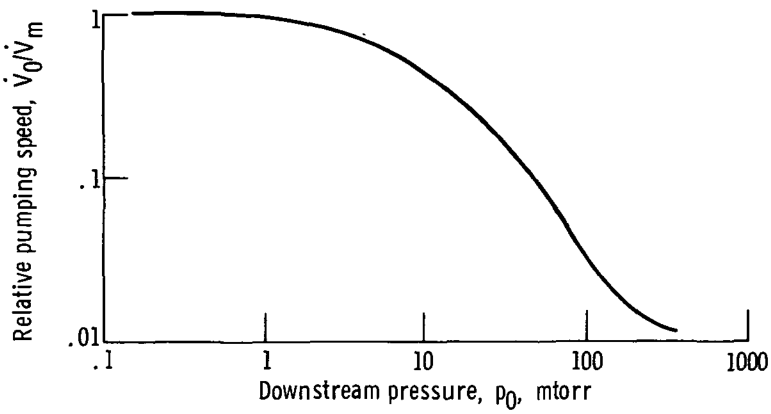

Figure 2. - Typical relative volumetric pumping speed of diffusion-pumped system. ($\dot{V}_m$ is the maximum pumping speed.)

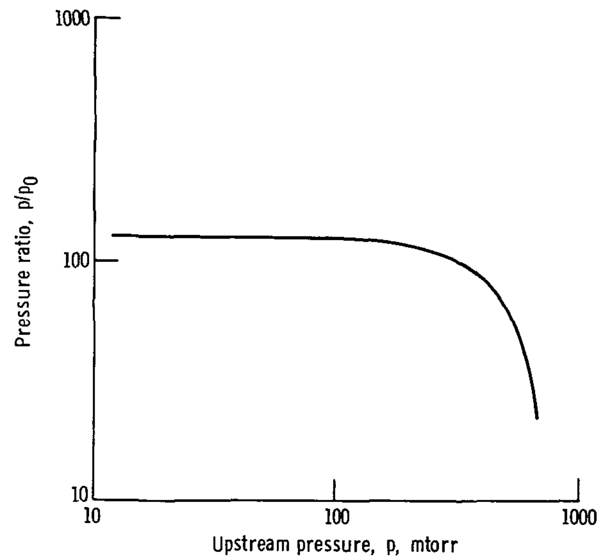

Figure 3. - Pressure ratio to be expected from system of figure 2 if $\dot{V}_m/G_a$ = 125. (See eq. (3).)

$$p = \left(1 + \frac{\dot{V}_m}{G_a} \cdot \frac{\dot{V}_0}{\dot{V}_m}\right) p_0 \tag{3}$$

allows construction of a graph of expected p/p_0 versus p for a system that follows figure 2. In equation (3), $\dot{V}_m$ is the maximum value attained by $\dot{V}_0$. The form of this relationship is shown in figure 3, for the case where $\dot{V}_m/G_a = 125$. The quantity $1 + \dot{V}_m/G_a$ represents the pressure ratio attainable when maximum pumping speed is available.

The curve of figure 3 may not be extrapolated indefinitely to the left because the additional phenomena of outgassing and permeation become prominent at very low pres-

sures. These effects were treated in reference 2.

Dynamic relations. - The time constant of response of upstream pressure p to an abrupt change of leak valve setting or to an abrupt change of gas-inlet pressure is

$$\tau = \frac{V}{G_a} \tag{4}$$

where V is upper-chamber volume. The value of τ is important in establishing the speed with which a multipoint calibration can be performed. The experimental determination of τ in the free-molecule regime also provides a means of estimating the annular clearance b between piston and orifice because annulus conductance is then given fairly accurately by

$$G_a = \pi D b \cdot \frac{v_a}{4} \tag{5}$$

where D is orifice diameter and v_a is arithmetic-mean molecular speed.

PRACTICAL DESIGN

Downstream Pressure Measurement

Pressure in the plenum on the downstream side of the piston may be measured with a calibrated thermal-conductivity gage when the pressure exceeds about 5 millitorr and with a conventional triode ionization gage when the pressure is less than 5 millitorr. Other gages that may be used are the high-pressure triode ionization gage (ref. 4) and the capacitance diaphragm gage.

Orifice

The baseplate orifice may conveniently be the existing port of the vacuum-system baseplate, provided the cylindrical walls of the port are adequately straight, round, and smooth; port area should be on the order of 100 square centimeters. The vacuum system used to test the piston manometer described here happened to have an orifice area of 74 square centimeters.

Piston

The piston used in the present tests was a cold-rolled stainless steel disk (0.025 mm thick), machined to an accurate circular planform whose diameter was 0.09 millimeter less than the orifice diameter ($\approx$10 cm). The machining was performed by clamping the sheet between two steel plates and then turning the assembly. Thereby, piston diameter was equal to the diameter of the steel plates, as measured by a micrometer. Piston mass was about 1.5 grams.

To support the piston, three holes (0.3-mm diameter) were punched through the sheet, 120° apart (at about a 34-mm rad.) and 0.25-millimeter-diameter soft constantan wires were passed through the holes (see fig. 4). The lower ends of the wires were twisted into loops and soldered. A washer between the soldered loop and the disk prevented jamming of the wire in the hole.

The supporting wires (about 40 cm long) were clamped at their upper ends to a central block attached to the dynamometer. The long length of the suspension resulted in only a small horizontal force between the edge of the piston and the cylindrical wall of the orifice. When there is flow through the annulus, the piston swings freely in the orifice, so that vertical friction also is negligible.

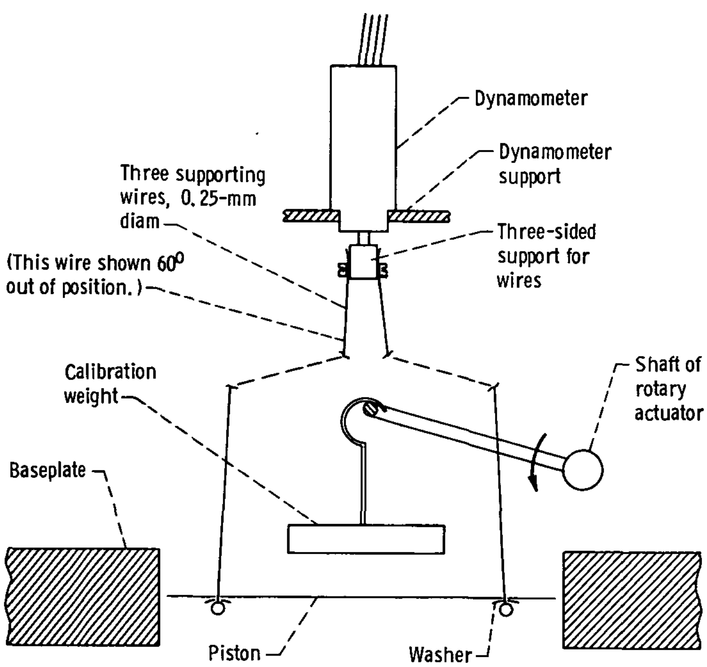

Figure 4. - Piston assembly and installation.

Dynamometer

The dynamometer was a commercial unbonded-strain-gage force transducer with a nominal range of 0.6 newton (60 grams force) and a deflection at full scale of 0.12 millimeter. Natural mechanical vertical frequency of the assembly, with suspended piston, was 90 hertz. Cable cover, shielding, and excess insulation were removed from the leads in order to minimize outgassing and virtual leaks.

The 350-ohm, four-active-arm bridge was continuously energized at one-tenth of its normal supply voltage in order to reduce excessive temperature rise of the unbonded wires in the vacuum environment. Total power dissipation was 0.7 milliwatt. Full-scale output was 7.5 millivolts. Input power to the bridge was derived from a regulated direct-current power supply and delivered through a reversing switch. Use of this switch permitted the elimination of the effect of thermal voltages in measurement of bridge output voltage. Output voltage was measured on a continuously connected integrating digital voltmeter with 1-second integration period and least count of 1 microvolt. One microvolt was equivalent to about 0.08 millitorr.

In Situ Calibration System

A 50-gram dead weight, calibrated to class S tolerance (0.03 percent), was suspended from a radial arm attached to the shaft of a bellows-sealed rotary actuator, so that the weight could be placed on top of the piston at will. The placement and removal of this weight permitted determination of dynamometer sensitivity at any time.

Operational Procedures

The operational procedure was intended (a) to measure the ratio between the calibration weight and the net force on the piston, so that the dynamometer would serve only as a transfer instrument, and (b) to cancel effects of thermal voltages when dynamometer output voltage was measured.

Pressure upstream of the leak valve (fig. 1) was maintained by a regulator at about 1.2 bar. The calibration procedure entailed the following steps.

(1) The leak valve was opened to provide the desired reading of the pressure gage to be calibrated. The gage reading was recorded only after a time equal to about 7 τ (eq. (4)) had elapsed. (In the present apparatus τ was about 60 seconds.)

(2) The output voltage of the dynamometer was read before and after operation of the input reversing switch.

(3) To create the condition $\Delta F = 0$, the downstream high vacuum valve was closed,

and the leak valve was closed immediately thereafter, before upper-plenum pressure could change more than 10 millitorr. After a time equal to about $7\alpha\tau$ (where α is the ratio of downstream-plenum volume to upstream-plenum volume and is ordinarily a small fraction), the output voltage of the dynamometer was read before and after operation of the input reversing switch.

(4) The calibration weight was lowered onto the piston, and the output voltage of the dynamometer read before and after operation of the input reversing switch.

(5) The calibration weight was lifted off the piston, the high-vacuum valve was opened, and the leak valve was immediately re-opened and set to provide the next desired calibration pressure.

If w is the weight of the calibration weight and if e_j^+ and e_j^- are the two digital-voltmeter readings in step j above ($j = 2$, 3, or 4), then the differential force across the piston at the end of step (1) is

$$\Delta F = w \cdot \frac{(e_2^+ - e_2^-) - (e_3^+ - e_3^-)}{(e_4^+ - e_4^-) - (e_3^+ - e_3^-)} (1 + \beta) \tag{6}$$

where $\beta = \beta(\Delta F/w)$ is the nonlinearity correction for the dynamometer ($\beta = 0$ for $(\Delta F/w) = 0$ or 1, and is a maximum at $(\Delta F/w) \approx 1/2$).

The averaging of the reversed-polarity readings in steps (2) to (4) eliminated the effect of thermal voltages in the detector circuit. The use of the $\Delta F = 0$ reading (step (3)) and the calibration-weight reading (step (4)) at each calibration point eliminated the effects of progressive shift in dynamometer sensitivity and in dynamometer zero with time or with the level of upper-plenum pressure. The dynamometer was used solely as a transfer instrument rather than as an absolute force-measuring device. From repeated measurements, it was determined that the probable errors of a single determination of zero and of sensitivity (slope) by the reversed-polarity technique were 0.03 millitorr and 0.02 percent, respectively.

The use of the above procedure resulted in a calibration interval of about 15 minutes per point with the present apparatus.

TEST RESULTS AND ANALYSIS OF ACCURACY

Accuracy of pressure determination will depend on the accuracy with which each of the terms in equation (1) can be measured and on the relative importance of these terms. A given fractional error in area A or net force ΔF will produce almost the same

fractional error in pressure p. However, a given fractional error in pressure p_0 will be attenuated by a factor equal to the pressure ratio.

Piston

Piston diameter may be measured directly with an optical projector or measuring microscope or indirectly by using a micrometer to measure the diameter of the finished steel plates between which the stainless-steel sheet is clamped for machining purposes. The resultant uncertainty in piston area, including the effect of imperfect correction for thermal expansion, is less than 0.03 percent.

Dynamometer

To determine the nonlinearity correction β (eq. (6)), the dynamometer was calibrated with class S weights. The load-application directions (load increasing or load decreasing) in which the loads were applied and removed were the same as the corresponding load-application directions described under Operational Procedures, so that the effects of systematic hysteresis were corrected for. The maximum value of the nonlinearity correction was 0.1 percent.

It is of incidental interest that, as upper-chamber pressure p was changed from 1 to 500 millitorr, the sensitivity dropped 0.8 percent and the zero shifted 2 percent of full scale; this zero shift is equivalent to 11 millitorr. There was very little additional change between 500 millitorr and 1 bar. The changes varied systematically with p. Although these shifts are not related to the present usage of the piston manometer (as described under Operational Procedures), their systematic nature suggests that, if it were desired to shorten calibration time at the expense of accuracy or to follow a changing bell-jar pressure, the dynamometer could be used as an absolute-force-measuring device, if systematic corrections were applied. However, the corrections, especially the zero correction, would probably differ considerably for gases of considerably different thermal conductivity, since the shifts are believed principally due to changes in temperature of the unbonded-strain-gage wires. The shifts quoted above are for nitrogen.

To reveal any statistically random error, of undefinable origin, that might be associated with the ΔF-measuring technique, a capacitance diaphragm manometer was connected between the upper and lower plenums, and a calibration performed as upper-plenum pressure was increased in stepwise fashion from 1 to 500 millitorr. Diaphragm manometer readability was 0.1 millitorr below about 100 millitorr and 0.3 millitorr at higher pressures. True differential pressure, computed as $\Delta F/A$, was compared with the diaphragm manometer indication, Δp. This comparison eliminated the downstream pres-

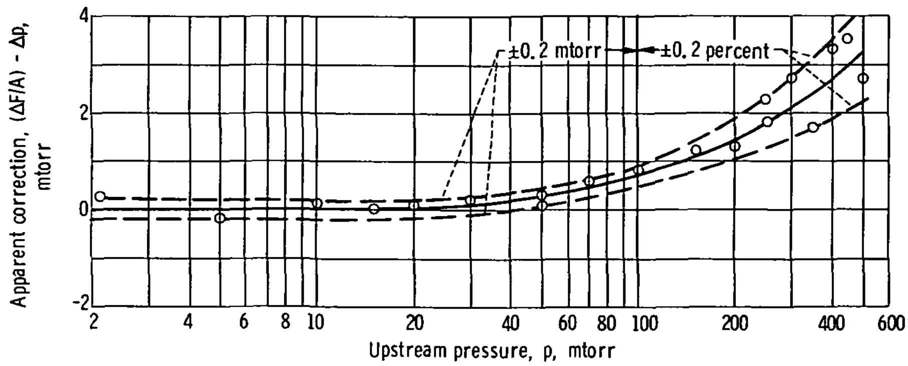

Figure 5. - Difference between piston and diaphragm manometers.

sure p_0 from the calculations. The difference between indications of the two manometers is plotted in figure 5. The shape of the mean curve is immaterial; only the random variations of individual points from the mean are of interest. The average deviation from the mean curve is 0.1 millitorr for $p < 100$ millitorr and 0.1 percent for $p > 100$ millitorr. Since the variance of the difference between the two manometers is equal to the sum of the variances caused by each, these values are upper bounds on the random error due to the piston manometer technique.

Boundaries representing deviations of 0.2 percent (for $p \geq 100$ mtorr) and 0.2 millitorr (for $p \leq 100$ mtorr) are also shown in figure 5.

Downstream Pressure

No experiments were conducted to establish the accuracy of downstream-pressure measurement; reliance was placed on the estimate of this accuracy provided by previous work.

References 1 and 5 to 7 have indicated that the calibration of a calibrated ion gage will remain invariant with a probable error of 3 percent, presumably only if the gage has not been exposed to a highly active gas like oxygen. In hitherto unreported work, the author has confirmed that a conventional triode gage can yield comparably small inaccuracy at pressures up to 5 millitorr. This determination was made by use of a piston manometer similar to that described in reference 2 except that the upper limit of the dynamometer range was 5 millitorr. Because of their similarity, it is likely that high-pressure ionization gages, such as those of reference 4, would have comparable probable errors.

Reference 8 has shown that a thermal-conductivity gage of the Pirani type, with adequately precise secondary electrical circuitry and with controlled ambient sink temperature, is also capable of probable errors of less than 3 percent.

Reference 5 has reported that a capacitance diaphragm gage had a probable error of about 3 percent in the range above 3 millitorr provided that the reading at zero differential pressure could be set to zero.

Thus, under favorable circumstances and by use of calibrated gages, downstream pressure is measurable with a probable error of 3 percent.

If an uncalibrated ion gage is used, but the average calibration is known for the particular gage model, the experience reported in reference 1 indicates that the probable error in downstream pressure is about 10 percent. Similar data are not available on thermal-conductivity gages.

On the other hand, if data on the gage model are not available and reliance must be placed on the manufacturer's control-unit calibration, probable errors may become unacceptably large. Calibrations of conventional-triode ion gages, performed with a piston manometer, indicated that the published sensitivity factor may be in error by 50 percent. Calibrations of thermal-conductivity gages, performed by R. Holanda (private communication) with the volume-ratio apparatus described in reference 8, show that the probable error averages about 10 percent, in the range 5 to 10 millitorr, provided the zero-pressure indication has been correctly set. Such an adjustment is feasible in the normal use of the piston manometer by merely closing the leak valve.

In the determination of the random error of the piston manometer by comparing it with the diaphragm manometer, calibrated downstream gages were not used, since the downstream gage did not enter into the comparison. Downstream gages were used, however, to provide a rough indication of pressure ratio; their probable error is estimated to be 10 percent.

The percentage errors quoted for downstream-pressure measurement should be divided by the estimated pressure ratio to estimate the effect on upstream-pressure measurement.

Pressure Ratio and Pumping Speed

The comparison between piston and diaphragm manometers, intended to prove the dynamometer and the operational technique, was performed with a 10-centimeter water-baffled diffusion pump. The characteristics of this pumping system at the upper end of the useful pressure range determined the shape of the pressure-ratio curve $(p/p_0)(p)$ shown in figure 6.

The clearance between piston and orifice was measured directly with a wire feeler gage and was also estimated by application of equations (4) and (5). Both determinations led to the values $b = 0.055$ millimeter, $G_a = 2.0$ liters/sec. The pressure ratio p/p_0 averaged about 125 at $p_0 < 10$ millitorr. These measurements, inserted into equation (3),

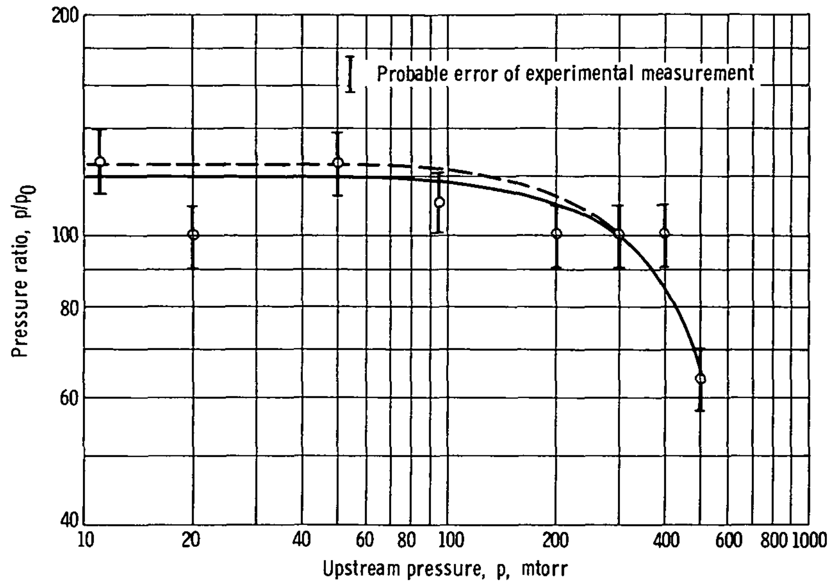

Figure 6. - Pressure ratio achieved in test system. Vertical lines indicate probable error of experimental measurement.

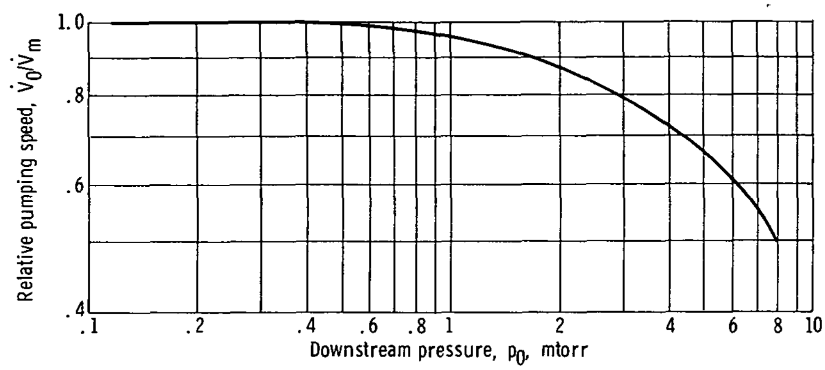

Figure 7. - Relative volumetric pumping speed of test system.

lead to an estimate of $\dot{V}_m$ as 250 liters/sec. The resultant curve of $(\dot{V}_0/\dot{V}_m)(p_0)$ is shown in figure 7. The range of downstream pressure shown in figure 7 is correlative to the range of upstream pressure shown in figure 6. Figure 7 was derived from figure 6 by application of equation (3). Vertical lines in figure 6 indicate the probable errors associated with the measured pressure ratios. The mean curve through measured points (solid line) differs from the curve (dashed line) computed from equation (3) by 5 percent.

Summary of Errors

A concise summary of errors is presented here. The probable error e_p is the

error that was exceeded half the time; the definition is independent of the nature of the error distribution. For a uniform distribution, the maximum possible error is $2e_p$; for the truncated quasi-Gaussian distributions common in engineering practice, errors exceed $2e_p$ less than 10 percent of the time.

Source	Errors
Piston area, A	Negligible
Net force, ΔF:	
Calibration weight	Negligible
Electrical circuit:	
Zero	$e_p = 0.03$ mtorr
Slope	$e_p = 0.02$ percent
Dynamometer:	
Nonlinearity	Max. = 0.1 percent
Random errors	$e_p = 0.1$ mtorr for $p \leq 100$ mtorr $e_p = 0.1$ percent for $100 < p < 500$ mtorr
Downstream pressure, p_0:	
Calibrated gage	$e_p = 3$ percent of p_0
Uncalibrated gage of a calibrated model	$e_p = 10$ percent of p_0
Manufacturer's calibration	$e_p = 10$ percent of p_0 Max. (1 case) = 50 percent of p_0

The effect of downstream pressure is attenuated by a factor equal to the pressure ratio. In the present tests p/p_0 exceeded 100 for $p < 300$ millitorr and was about 60 at $p = 500$ millitorr.

The combination of the enumerated errors leads conservatively to the representation of the overall probable error of the piston manometer as ±(0.2 millitorr + 0.2 percent).

DISCUSSION AND CONCLUDING REMARKS

Accuracy of pressure determination will depend on the accuracy with which each of the terms in equation (1) can be measured. Accuracy of gage calibration will depend, additionally, on the extent to which pressure in the calibration volume is uniform and equal to the pressure measured by the piston manometer. Reference 2 discusses the problem of achieving an isobaric condition and the effects of permeation through gaskets, outgassing of surfaces, and reactions at heated surfaces. The problem, though serious in the pressure range considered in reference 2, was not considered serious in the thousand-fold higher pressure range considered in this report and, hence, was not investigated experimentally. The experimental investigation was confined to a study of the

dynamometer as a transfer device for net-force measurement.

By using a highly linear dynamometer and a force-transfer technique in which only dynamometer linearity, rather than dynamometer accuracy, is important, the error of differential pressure measurement, including statistically random errors, can be made acceptably small, with probable errors on the order of 0.1 millitorr or 0.1 percent. More serious errors in determining pressure p can arise from inaccuracies in downstream-pressure measurement, unless the downstream gage has been individually calibrated and has retained its calibration. This dependency of p upon p_0 derives from the relatively low pressure ratio achieved in the present tests, compared with the pressure ratio obtained in reference 2 or the pressure ratio to be expected from equation (2) and the ratio $\dot{V}_0/G_a$. Higher pressure ratios could be achieved by increasing the diameter of the downstream plenum and using a pumping system that has higher pumping speed, particularly at high pressures. Reduction of dependence on downstream-pressure-gage accuracy, through a higher pressure ratio, is a desirable improvement. Nevertheless, when the downstream gage has a probable error of 10 percent, even a pumping system such as that represented by figure 7 provides a probable error on the order of $\pm$(0.2 millitorr + 0.2 percent) in the establishment of upstream pressure in the range $2 \leq p \leq 500$ millitorr.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 1, 1972,
502-04.

APPENDIX - SYMBOLS

A piston area

b annulus width

D orifice diameter

e_2 digital voltmeter reading in operational step 2

e_3 digital voltmeter reading in operational step 3

e_4 digital voltmeter reading in operational step 4

e_j digital voltmeter reading in operational step j ($j = 2, 3, 4$)

e_p probable error

ΔF net force

G_a annulus conductance

M molecular weight

p upstream pressure

p_0 downstream pressure

Δp pressure difference, $p - p_0$

R_0 universal gas constant

T absolute temperature

V upstream plenum volume

$\dot{V}_0$ volumetric pumping speed in downstream plenum

$\dot{V}_m$ maximum value of volumetric pumping speed $\dot{V}_0$

v_a arithmetic mean molecular velocity

w weight of calibration weight

α ratio of downstream-plenum volume to upstream-plenum volume

β nonlinearity correction

τ time constant

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